LEAK RATE AND LOCATION ANALYSIS THROUGH SLITS AND CRACKS IN PIPES BY NANO POROUS CERAMIC HUMIDITY SENSORS

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Abstract: Nano porous sol gel thin film humidity sensor was studied using commercially available reference humidity sensor from Honeywell Corporation. The main advantages of our developed set up for humidity measurements is low cost and high resolution yielding a full set of information on the variation of humidity at 250°C. Humidity is considered to be one of the most effective indicators of the leakage. On this ground we developed nano porous sensor which can be used for LBB (Leak before break) application. The ceramic sensors, based on sol gel thin film are shown to increase its capacitance upon water adsorption over the temperature range upto 250°C. The variation of capacity to voltage is shown to provide useful information on both break rate and location. The sensor installation spacing on the outer surface of the piping is determined as a function of the detection sensitivity. In this paper we have summarize the results of the development and characterization of ceramic humidity sensor for leak rate and location analysis by a microcontroller device.

Index Terms: Nano porous H. sensor, Linearization, Microcontroller circuit, Leak location.

1. INTRODUCTION

Humidity measurements have become increasingly important, in the industrialized world since it has been recognized that humidity has a significant effect on quality of life, quality of product, safety, cost, and health. This has bought about a significant increase in the humidity measurement applications and concurrent with this, an increase in research and development activities to improve measurements technique, accuracy and reliability of instrumentation.

Despite the increase in research and development activities during the last two decades to improve humidity sensors, the present state of art is still such that the humidity measurements required more care, more maintenance and more calibration than other analytical instruments. Furthermore there is still no sensor available that can even come close to covering the full dynamic range of water vapor levels. For this reason, many different measurement methods and sensors have been developed through the years, each having certain advantages and limitations and suitable for some, but not all applications. For some one not skilled in the field of humidity, it is in many case very difficult to make an intelligent choice of sensor, and when this is not done disappointing results will often occur.

The capacitive type sensor makes use of the increase of apparent dielectric permittivity due to moisture absorption. If therefore towards pollution and is suited for environment assessment. Most advanced humidity sensors on the market today are the “bulk polymer sensors”, consisting of a miniature electrode base plate coated with a humidity hygroscopic macro polymer. An electrical grid structure is vapor deposited upon the element and an electrical measurement is made which is a function of relative humidity. Because of the good chemical stability of polymer sensors, the sensors exhibit excellent resistance to most solvents and are therefore often used for individual applications, except for those having high concentrations of corrosive chemicals. Many common substances such as petrochemicals have little effect. Some different polymer are used, sensitivity to contaminants cause greater adverse effect on resistive sensors which others effects on resistive sensors which others effect capacitive sensors more. Also there is limitation of using polymers at high temperature. Hygroscopic ceramic nano porous thin aluminum oxide has been used which is superior in long term stability and sensitivity upto 300ºC is emphasized in their field of applications. Recent work has, therefore, concentrated on the hyper thin film ceramic nano porous humidity sensors [1-3].

A digital hygrometer describe in this paper also used a ceramic capacitor as the sensing element. Its capacitance changes non-linearly with relative humidity (RH) and temperature. Temperature dependent is small compared to dielectric sensitivity i.e. capacitance changes due to RH. The capacitance change should be detected in a digital form for linearization. The simplest circuit to perform such a function is a monostable oscillator. To encode only the capacitance change into a digital form, the dry capacitance when RH=0 is cancelled. Finally, a prototype hygrometer based on these techniques will described for use in leak detection.

2. SENSOR AND ITS FABRICATION PROCESS

The fig-1 shows the structure of the sensor. Its fabrication process is as follows.

Fig-1: Picture of the Nano Porous Thin Film Sensor

The Alumina sol solution was mixed with a calculated amount of PVA (Poly Vinyl Alcohol) in hot condition for 2 hours. The solution was made 100% bubbles for thin film preparation. Films coated on a gold coat α-Alumina Substrate of size (10 mm x 20 mm x 1 mm). Second electrode is formed on film coated substrates. It is then finally fired at

950°C for cuing the electrodes (Fig-1). The following procedure was adapted for preparation of films. The binder mixed sol was important for reproducible film. The films were deposited on gold coated on α-alumina substrate by dipping them in the prepared sol then pulling out with a speed of 10 cm/min 4 times with sol of Higher Surface tension. This was followed by drying and then sintering the films between 450°C – 500°C for a period ranging from 4 to 5 hrs. The thinner oxide layer resulted in much higher capacitance changes because capacitance is inversely proportional to the distance between two electrodes. The unique nano pore geometry for the sol-gel γ-Al_2O_3 thin film enhanced the entrapment of water molecules. Sol-gel γ-Al_2O_3 sensors therefore responded several times faster than the conventional sensors. As the pores were uniform through the area the sensors need not require any external heat. The sensor can be quickly dried to place it in a drying agent or by placing it in a dry air or nitrogen gas. The film was covered by gold electrode. The electrode was made perforated net structure for easy entry of water molecules into the substrate.

3. SENSOR CHARACTERISTICS AND SENSING PRINCIPLE

The sensor characteristic for different %RH and different film thickness are tabulated in table 1-3. The corresponding graph of the sensor is shown in fig-(2).

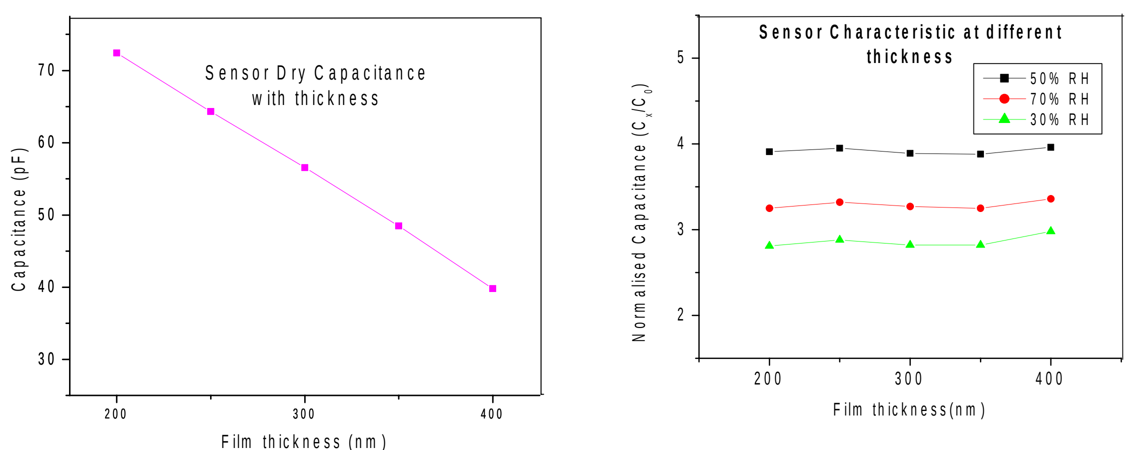

Fig-2(a): Dry Capacitance With Film Thickness

Fig-2(b): Normalized Capacitance With Film Thickness

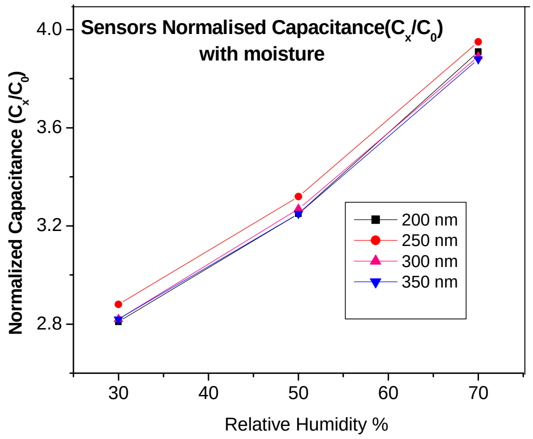

Fig-2(c): Normalized Capacitance (C_x/C_0) With Relative Humidity

Table-1: Sensor Characteristic At 30% RH

Sensors	Film Thickness (nm)	Dry Capacitance C_0 (pF)	Normalized Capacitance C_x/C_0 (pF)	Condition	
				RH%	Temperature
A	200	72.43	2.81	30	$28^{\circ}C$
B	250	64.32	2.88	30	$28^{\circ}C$
C	300	56.54	2.82	30	$28^{\circ}C$
D	350	48.46	2.82	30	$28^{\circ}C$
E	400	39.78	2.98	30	$28^{\circ}C$

Table-2: Sensor Characteristic At 50% RH

Sensors	Film Thickness (nm)	Dry Capacitance C_0 (pF)	Normalized Capacitance C_x/C_0 (pF)	Condition	
				RH%	Temperature
A	200	72.43	3.25	50	$28^{\circ}C$
B	250	64.32	3.32	50	$28^{\circ}C$
C	300	56.54	3.27	50	$28^{\circ}C$
D	350	48.46	3.25	50	$28^{\circ}C$
E	400	39.78	3.36	50	$28^{\circ}C$

Table-3: Sensor Characteristic At 70% RH

Sensors	Film Thickness (nm)	Dry Capacitance C_0 (pF)	Normalized Capacitance C_x/C_0 (pF)	Condition	
				RH%	Temperature
A	200	72.43	3.91	70	$28^{\circ}C$
B	250	64.32	3.95	70	$28^{\circ}C$
C	300	56.54	3.89	70	$28^{\circ}C$
D	350	48.46	3.88	70	$28^{\circ}C$
E	400	39.78	3.96	70	$28^{\circ}C$

The capacitance of the RH sensor measured at 1 KHz is plotted in fig-2 as a function of RH and temperature. The abscissa represents the RH measured by commercial dew point meter (SHAW) of accuracy ±0.1ºC and the ordinate is the sensor capacitance C_x normalized by its dry capacitance C_0 when RH=0. C_0 is typically 70pF with a film thickness of 200 nm. The temperature dependence of C_0 is very small and it is neglected in the following discussions. Table one indicates fractional capacitance change at different film thickness.

The comparison shows that the fractional capacitance changes of the ceramic based RH sensors are almost independent of their fabrication process and film thickness. The response time for the step change between 40 and 80% change is shorter than 12 s. these facts indicate that the capacitance change of sensor can be explained by the increase in the apparent dielectric constant ε_s of the hygroscopic film due to physical sorption of water vapour. Absorbed water can be regarded responsible as being distributed uniformly throughout the film because the fractional capacitance change is independent of film thickness. Therefore the sensor capacitance C_x is given by

$$C_x / C_0 = L_p / \in_p \left(\int_0^{Lp} \frac{dl}{\in_s} \right) = \frac{\in_s}{\in_p}$$

Where ε_p is the dielectric constant of the alumina thin film (ε_s= 8) and Lp is the thickness of the hygroscopic film.

The apparent dielectric constant ε_s of the hygroscopic film is given by Looyengas empirical equation [17].

$$\in_s = \left\{ \gamma \left(\in_w^{1/3} - \in_p^{1/3} \right) + \in_p^{1/3} \right\}^3 \quad \text{...(1)}$$

Where γ is the fractional volume of water in the film and ε_w is the dielectric constant of water given by [16]

$$\in_w = 78.54\left\{1 - 4.6\times10^{-4}(T-298) + 8.8\times10^{-6}(T-298)^2\right\} \quad \text{.............................(2)}$$

Where T is the temperature in Kelvin. The fractional volume γ can be obtained from the measured capacitance C_x using (1) and (2).

4. LEAK LOCATION WITH MICROCONTROLLER DEVICE

In order to reduce the construction cost and radiation exposure associated with maintenance of piping restraints, local humidity sensors are recently used for high sensitivity and accuracy of leak location [5-7, 10-15]. Such a humidity based local sensor suggested in Reg. Guide [4] 1.45 is already implemented in European power

Plants [8,9]. On this ground the requirements of leak sensors according to NUREG-106[4] are as follows-

1) Leak detection system should be able to detect 1gram per minute (gpm) in the identified leak, and 0.1 gpm in the unidentified leak

2) The leak detection systems must have three diversity, each with three redundancies.

3) Local sensors are applicable.

4.a) Block diagram of location analysis circuit and its operation

The block diagram of the location analysis circuit is shown in fig-3(a) and also the schematic diagram is shown in fig-3(b). The heart of this circuit is 8051 based microcontroller (AT89C52).

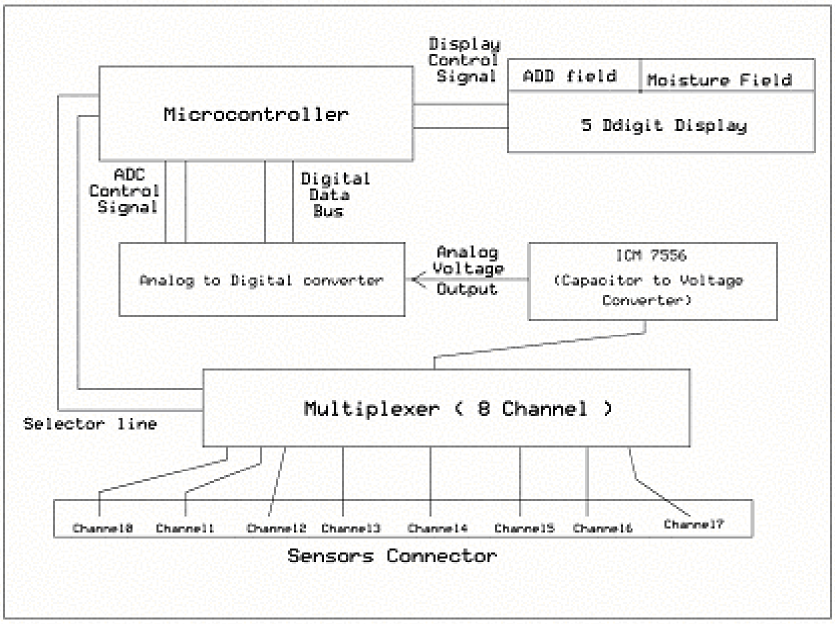

Fig-3(A): Block Diagram Of Location Analysis Circuit.

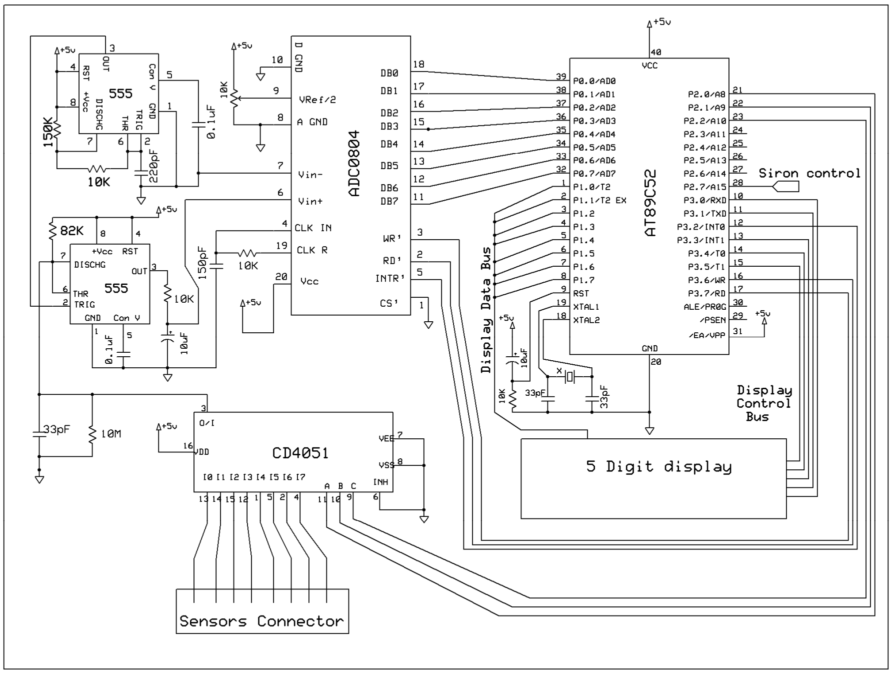

Fig-3(B): Schematic Diagram Of The Location Analysis Circuit.

The sensors are mounted on the surface of the stream pipe and the terminals from the sensors are connected in the input of analog multiplexer. The microcontroller select one sensor at a time by providing multiplexers' selector input and read corresponding moisture around that sensor with the help of ICM7556 and ADC. After that it checks the moisture level for the boundary limit of the moisture. If this moisture level crosses the boundary level it gives an alarm and displays the corresponding sensor location in the address field of the sensor and moisture value in the moisture field.

5. ANALYSIS OF LEAK DETECTABILITY

LEAKAGE DISTRIBUTION

Humidity response test results show that nanoporous $\alpha-Al_2O_3$ sensor can detect humidity at 300˚C. Local humidity monitoring around the pipe can be a sensitive and rapid method to detect high temperature steam leakage.

Static characteristic of monolith microporous and thin film nanoporous sol gel processed $\alpha\text{–}Al_2O_3$ sensors for good sensitivity and compatibility with high temperature (250˚C). Test were performed with the specimen initially in dry and then humid air to compare the humidity response characteristic, from the equation $\varepsilon^* = \varepsilon - j\varepsilon = \varepsilon - j\left(K \big/ \omega\varepsilon_0\right)$. The overall capacity changes to a high value for nano porous material and microporous. Nano porous material is sensitive to trace moisture level and it is also sensitive to trace leakage in 45 seconds whereas microporous materials takes 315 seconds to response. The responses of microporous and nanoporous sensors with time as shown in fig-4(a) and fig-4(b) respectively.

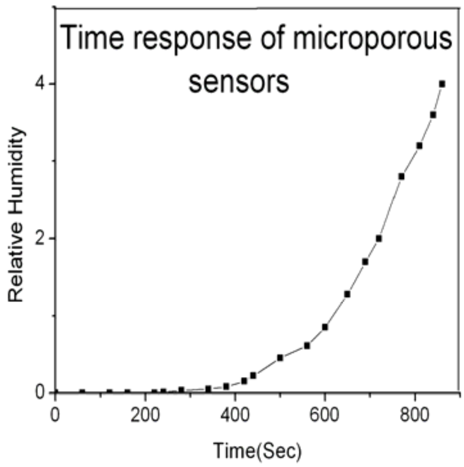

Fig-4(a): Response of microporous sensor with time.

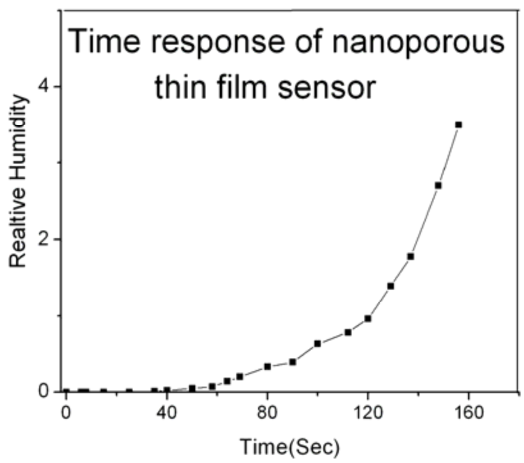

Fig-4(b): Response of nanoporous sensor with time.

The water vapor in the annulus pipe between main stream line and insulation sheet is assumed to migrate by diffusion only. The convective mass trabport will indicate detection sensitivity. More conservative estimate can be done by following one dimensional diffusion analysis for the pipe where moisture flows in longitudinal direction of X axis as follows.

Relative humidity = Vap. Pressure/ Saturated V_p = $({}^{n}H_2O\ R_T/V)/P_{Sat}$ ……………………..(3)

${}^{n}H_2O(x,t) = g_s I\sqrt{(t/{}^{D}H_2O)}\ \exp(-x^2/4{}^{D}H_2Ot)-(x/2DH_2O)[1-\mathrm{erf}(x/2\sqrt{({}^{D}H_2O)}]$ ……….(4)

The above equations represent relative humidity as a function of distance from leak location and time.

6. LEAKAGE LOCATION ANALYSIS

From previous discussion we see nanoporous sensors have better response to the moisture. Hence for location analysis, all the sensors used are of nanoporous type. Picture of the experimental location analysis circuit during testing at laboratory scale is shown in fig-6(c). The experimental set-up for detection of leakage location of a stream carrying pipe from side view is shown in figure-6(a) and the cross sectional view is shown in fig-6(b). We consider the stream pipe of 18 meters in length and we mount eight nos of high temperature thin film moisture sensors perpendicularly with the surface of the pipe. Separation between the two successive sensors is about 2 meters. Sensors are addressed as “000” to “007” from stream inject side to stream out late side. After mounting the sensors, these are covers with the insulating material and connections from the sensors are taken outside the insulating cover. The microcontroller based control systems continue to check all the sensors and display the highest moisture level present in the surface region of the pipe and the nearest sensor location. If there is any leakage in the pipe the stream comes outside the pipe and moisture of that region increases. The nearest sensor first senses the moisture and the corresponding sensor location and moisture value is displays on the display board. All other sensor data are stored in the microcontrollers’ RAM area. This will be checked after the experiment. Also a siren is on and it continues if the moisture level is in high value range. Experimental results for four set up are summarized in the table-1 and also the response of the sensors for set-up-4 is shown in fig-5. All the tabulated data have been taken 1 minute from the time stream line open.

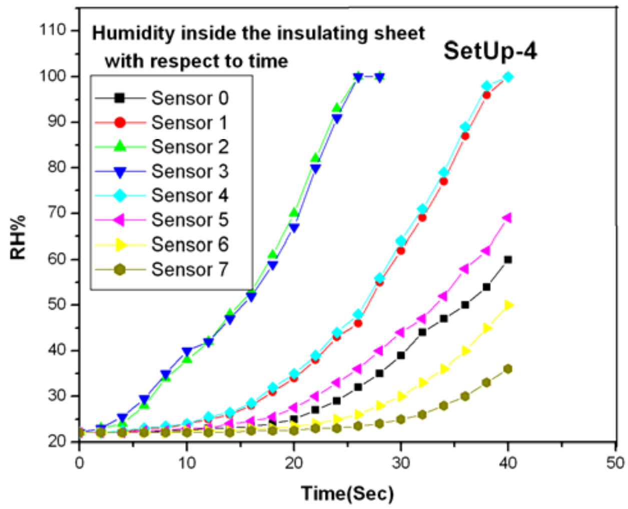

Fig-5: Response Of The Sensors For Set Up-4

Table-I: RH Of Different Sensor At Different Location Of Different Set Up

Set up-1		**Set up-2**		**Set up-3**		**Set up-4**	
Crack at 3.5 m from the stream in late		Crack at 0.5 m from the stream in late		Crack at 15.0 m from the stream in late		Crack at 7.0 m from the stream in late	
Sensors address	RH%	Sensors address	RH%	Sensors address	RH%	Sensors address	RH%
000	82	000	100	000	43	000	51
001	100	001	68	001	42	001	78
002	74	002	54	002	43	002	100
003	58	003	48	003	48	003	100
004	47	004	42	004	58	004	76
005	44	005	42	005	73	005	52
006	42	006	43	006	100	006	43
007	43	007	43	007	100	007	43

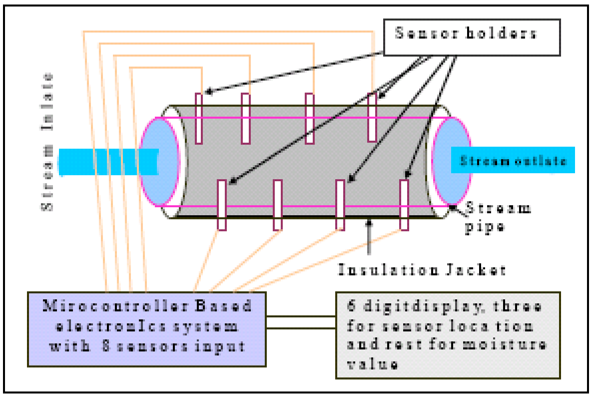

Fig-6(a): Block Diagram Of Experimental Set-Up For Detection Of Leakage Location

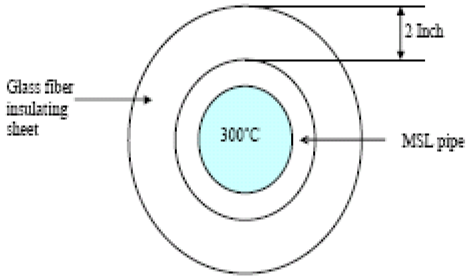

Fig-6(b): Front View Of Experimental Set Up

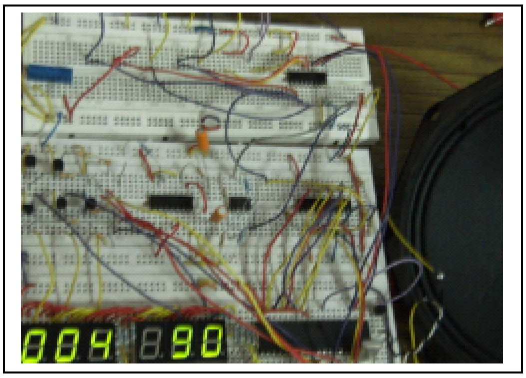

Fig-6(c): Picture Of Location Analysis Circuit During Test.

7. CONCLUSION

Ceramic humidity sensor has been developed to estimate leak rate and location through pipe cracks/slits in a pressurized system. The capacitive based sensor is shown to respond to humidity changes within 45 seconds at temperature of 300˚C. The transient response to AC is found to be useful for humidity detection. The input voltage generated by capacity to voltage converter is linearized by microcontroller device for exact location of cracks and leak rate in a pipe.

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